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(19)

(54) COMPOSITE INTERLAYER FOR LAMINATED SAFETY GLASS

(71) We, MONSANTO COMPANY, a corporation organised under the laws of the State of Delaware, United States of America, of 800 North Lindbergh Boulevard, St. Louis, Missouri 63166, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

The present invention relates to a composite suitable for use as an interlayer for laminated safety glass; and to a process for the preparation thereof. More specifically, the present invention relates to such a composite which contains a color gradient.

It is known to use thermoplastic polymeric material, in particular polyvinyl butyral, in sheet form as an interlayer for the laminated safety glass used in vehicles, especially windshields, and in architectural applications. In many applications the interlayer is tinted with an ink so as to provide a colored laminate. One of the major uses for tinted laminates is in automobile windshields. In these applications the interlayer is tinted with a color gradient which is positioned so as to form a glare-reducing band at the top of the laminated windshield.

The gradient printing operation for the interlayers, used in windshields, usually involves printing the ink in the form of dots on the surface of the sheet. The sheet is then dusted with a material such as sodium bicarbonate to minimize undesirable ink transfer. Prior to use as an interlayer, the sodium bicarbonate dust is washed off the sheet which is then dried and laminated to the glass. Printing the interlayer material gives rise to the need to dust and then wash the sheet. Moreover, the adhesion of the printed portion of the sheet to glass may be adversely effected by the printing step.

A definite need exists in the art for interlayer material having a color gradient with desired adhesion to glass and which does not require dusting and washing prior to use. Such a material is provided by the present invention the material being a composite comprising at least two sheets of thermoplastic polymeric material combined together in face-to-face contact with at least a portion of at least one of the sheets being printed with an ink on a side of the sheet that is in face-to-face contact with another of the said sheets, the thermoplastic polymeric material being polyvinyl butyral, a polyurethane, a polyamide, poly(ethylene-vinylacetate) or poly(ethylene-vinyl alcohol). The preferred thermoplastic polymeric material is polyvinyl butyral, and the specification is written with particular reference to this preferred embodiment.

The invention also comprises a process for preparing a composite in accordance with the invention, which process comprises:

A. passing a first continuous sheet of thermoplastic material which is polyvinyl butyral, a polyurethane, a polyamide, poly(ethylene-vinyl acetate) or poly(ethylene-vinyl alcohol) between nip rolls to impart machine direction, sheet tension and drawdown into the sheet;

B. adjusting the temperature of the sheet to a temperature in the range of from 32 to 82°C;

C. removing any wrinkles from the sheet; and then

D. combining in face-to-face contact the first continuous sheet with a second continuous sheet of thermoplastic material which is polyvinyl butyral, a polyurethane, a polyamide, poly(ethylene-vinyl acetate) or poly(ethylene-vinyl alcohol) wherein the second sheet has received substantially the

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same treatment as outlined in process steps A to C above, at least a portion of at least one of the sheets being printed with an ink on a side of the sheet that is brought into face-to-face contact with the other sheet; and

E. cooling the resulting composite.

In a further embodiment, the invention provides a process for preparing a composite suitable for use as an interlayer for laminated safety glass, which process comprises combining a sheet of polyvinyl butyral which has been printed on both sides with a color gradient between two other sheets of polyvinyl butyral by passing the sheets through a combining nip; wherein the temperature, tension and line speed of each of the sheets, prior to entering the combining nip, is substantially the same.

The invention also comprises a process for preparing a composite suitable for use as an interlayer for laminated safety glass, which process comprises:

A (1) passing a first continuous sheet of polyvinyl butyral between a first set of nip rolls to impart machine direction, sheet tension and drawdown into the sheet;

(2) adjusting the temperature of the first sheet to a temperature in the range of from 32 to 82°C. while removing any wrinkles in the sheet;

(3) allowing the first sheet to assume a catenary profile between second and third sets of nip rolls;

(4) adjusting the temperature of the first sheet to a temperature in the range of from 32 to 82°C; and simultaneous with steps 1 to 4 above;

B (1) passing a second continuous sheet of polyvinyl butyral which is printed on one side with a color gradient, between a fourth set of nip rolls and

(2) allowing the second sheet to assume a catenary profile between fifth and sixth sets of nip rolls;

(3) adjusting the temperature of the second sheet to a temperature in the range of from 32 to 82°C; and

C (1) passing the first and second sheets into a combining nip where they are combined wherein the printed surface of the second sheet of polyvinyl butyral is combined to the first sheet so as to provide a composite wherein the bond strength between the first and second sheet is at least 0.295 Kg/cm;

(2) cooling the resulting composite;

wherein the process steps outlined in A and B above are controlled to provide substantially the same line speed, sheet tension and temperature in each of the first and second sheets of polyvinyl butyral being fed into the combining nip.

Figures I to III inclusive of the accompanying drawings illustrate cross-sections of the composites of the present invention.

Figure I illustrates a sheet of polyvinyl butyral 11 printed with an ink 12 wherein the printed side is in face-to-face contact with a second sheet of polyvinyl butyral 13.

Figure II illustrates two sheets of polyvinyl butyral 11, each of which is printed with an ink 12, wherein the printed sides are combined.

Figure III illustrates a sheet of polyvinyl butyral 11, printed on both sides with an ink 12, wherein the printed sides are combined to two other sheets of polyvinyl butyral 13.

Figure IV is a schematic diagram illustrating the extrusion of molten polyvinyl butyral onto the surface of a sheet of polyvinyl butyral which has been printed with a color gradient.

Figure V is a schematic diagram illustrating the combining of two sheets of polyvinyl butyral, one of which has been printed with a color gradient wherein the color gradient is at the interface of the resulting composite.

Figure VI is a schematic diagram illustrating another more detailed process for combining two sheets of polyvinyl butyral, one of which has been printed with a color gradient wherein the color gradient is at the interface of the resulting composite.

In general the polyvinyl butyral resins employed in the present invention have Staudinger molecular weights ranging from 50,000 to 600,000 and preferably from 150,000 to 270,000 and may be considered to be made up, on a weight basis, of from 5 to 30% hydroxyl groups, calculated as polyvinyl alcohol, 0 to 10% ester groups, calculated as polyvinyl ester, and the balance substantially butyraldehyde. The polyvinyl butyral resin will preferably contain, on a weight basis, from 9 to 25% hydroxyl groups, calculated as polyvinyl alcohol and from 0 to 3% acetate groups, calculated as polyvinyl acetate, the balance being substantially butyraldehyde.

It is conventional to adjust the alkalinity titer of the polyvinyl butyral resin so as to optimize the impact strength of the resulting laminate. Methods for adjusting the alkalinity titer are discussed in, for example, U.S. Patents Nos. 3,262,835, 3,294,490, 3,396,074, 3,271,235 and 3,231,461.

The polyvinyl butyral resin is usually plasticized with from 20 to 80 parts by weight of plasticizer per 100 parts by weight of resin and more commonly with between 30 and 50 parts by weight for normal windshield use. This latter concentration is generally used with polyvinyl butyrals containing 18 to 23% vinyl alcohol by weight. In general, the plasticizers

which are commonly employed are esters of a poly-basic acid or a polyhydric alcohol. Particularly suitable are triethylene glycol di(2-ethylbutyrate), dibutyl sebacate, and di(betabutoxyethyl) adipate. The resulting plasticized resin mixture is then generally extruded in the form of sheets which are then printed as described below.

The thickness of the individual polyvinyl butyral sheets used to prepare the composites of the present invention is most commonly in the range 0.127 to 1.524 mm with a range of from 0.127 to 0.762 mm being preferred for use in automobile windshields. The choice of thickness of the individual sheets will ultimately depend on the thickness desired in the composite sheet and those skilled in the art can readily select sheets of appropriate thickness for any given application. The total thickness of the composite can, for example, be within the range 0.51 to 1.91 mm.

In one embodiment of the present invention the surface of the sheet to be printed, which is the surface which will ultimately be in laminated contact with another sheet of polyvinyl butyral, is relatively smooth so as to provide a better surface for printing. On the other hand, the surface of the polyvinyl butyral sheet which will ultimately be in laminated contact with the glass member of the laminated safety glass will preferably have a conventionally rough surface so as to prevent blocking of the rolled sheet, and to facilitate de-airing of the laminate. The above mentioned surfaces are well known in the art and need no further discussion here.

The processes for tinting automobile windshield interlayer material are usually designed to give a color gradient. In the color gradient printing process the ink is printed in a pattern such that there is a dark section at the top of the interlayer which gradually and uniformly fades off into a clear section in the direction of the bottom of the interlayer. In the resulting laminated windshield the dark portion of the gradient is at the top of the windshield. In automobile windshields the darkest portion of the color gradient usually has an optical density in the range of from 1.70 to 0.70 which corresponds to a percent light transmission of from 2 to 20%, respectively. Preferably, the light transmission in the darkest portion of the color gradient is in the range of from 4 to 10%.

The present invention is also applicable to those polyvinyl butyral printing operations which do not require a color gradient. These usually involve non-windshield application wherein the sheet is uniformly tinted.

Referring to the drawings, Figure IV illustrates one embodiment of the present invention wherein molten polyvinyl butyral is extruded onto the printed surface of the polyvinyl butyral sheet. In Figure IV polyvinyl butyral sheet material 50 is passed over an idler roll 51 through a printing station 52 wherein one side of the sheet is printed. The printed sheet is then passed over a series of drive rolls 53 and idler rolls 51 to a nip formed by the outlet of the extruder 56 and a die roll 57 where molten polyvinyl butyral is extruded onto the printed surface of the polyvinyl sheet. The resulting composite is passed over the die roll 57, past a stripper roll 54 and a take-off roll 55 and an idler roll 51 to a winder 58. The resulting composite 59 is then transferred to storage or shipping.

Referring again to Figure IV, polyvinyl butyral sheet material may optionally be fed to the printing station directly from an extruder or other sheet making apparatus thereby eliminating the necessity of winding and unwinding the sheet material prior to feeding it to the printing station.

Figure V illustrates a sheet combining operation wherein two 15 mil sheets of polyvinyl butyral are combined to form a 30 mil composite having a printed color gradient at the interface of the composite sheet. The first sheet 60 is clear material having conventional surfaces on both sides while the second sheet 61 is printed on one side with a color gradient which is designed to appear as the tinted color band at the top of a windshield in the resulting laminate. Preferably, the printed side is relatively smooth while the opposite side of the sheet has a conventional rough surface.

The clear sheet 60 enters the top section of the combining unit through the upper tension rolls 62. These tension rolls serve two purposes: (1) they act as a positive, constant speed driving section; and (2) they are driven at a slower line speed than the combining rolls 65 to impart a desired amount of sheet tension and drawdown in the clear sheet 60 to prevent cross machine direction wrinkles and to control curl. This sheet tension is required to eliminate wrinkles in the combined sheet due to sheet growth during heating and to match the amount of drawdown in the upper clear sheet 60 to the drawdown in the lower printed sheet 61. The drawdown is usually determined by measuring the width change of the sheet. If the drawdown in the upper sheet does not match that of the lower sheet, the final product will have excessive curl. The upper tension rolls are run 1% to 30% slower than the combining rolls 65 with 4% to 16% slower being typical values.

The clear sheet 60 then passes under a bank of electric infra-red pre-heaters which

a sheet temperature in the range of 32°C adjust the temperature of the incoming sheet to a temperature in the range of from 32° to 82°C. The preferred temperature is 43° to 71°C, as measured with an infra-red pyrometer just after the bowed flexible rolls 63. After being pre-heated, the sheet passes over a flexible bowed roll 63 (Mt. Hope Vari-Bow Roll) which serves to spread the sheet in a cross-machine direction, eliminating any wrinkles which may have formed due to sheet growth during heating.

The clear sheet 60 is then carried to the upper lay-on roll 64 which transfers the sheet to the upper combining rolls 65. The lay-on roll 64 is operated at anywhere from zero nip pressure (nip open) to a nip pressure of 2.95 Kg/cm depending on the particular sheet used as well as other operating conditions. The clear sheet 60 wraps the upper combining roll 65 until it reaches the combining nip 69. The combining rolls are heated at from 32°C to 82°C with 43° to 71°C being preferred. The combining nip pressure runs from 1.48 Kg/cm to 8.85 Kg/cm with 2.95 to 5.90 Kg/cm being preferred. The upper combining roll 65 surface is slightly less tacky than the bottom combining roll 65 surface to facilitate sheet transfer to the bottom roll.
perature achieved in the top section. The printed sheet 61 is then transferred to the

Polyvinyl butyral sheet 61 printed with a color gradient enters the unit through the bottom relaxing nip 66, generally at a temperature of from 21° to 49°C with a drawdown of from 10% to 35%. The sheet is allowed to relax between the relaxing nip 66 and the lower tension nip 62 by running the lower tension nip 3% to 20% slower than the relaxing nip. The printed sheet 61 goes through the lower tension rolls 62, which are run at a line speed equal to or less than the combining roll line speed and preferably at the same line speed as the upper clear sheet 60. The lower tension rolls serve the same purpose as the upper tension rolls, namely they build in machine direction sheet tension to prevent cross-machine direction wrinkles and to control curl. The lower tension roll speed is from 1% to 30% less than the combining roll speed with from 4% to 16 % less being preferred.

The printed sheet 61 then passes under electric infra-red preheaters which are set to give temperatures equal to the tem-
lower combining roll 65 by the lower lay-on roll 64, operating in a manner similar to the upper lay-on roll. The sheet wraps the lower combining roll 65 which is heated to the same temperature as the upper combining roll.

After passing through the combining nip transferred to a cooling roll 67. The cooling roll surface is usually kept below 21°C. After leaving the cooling roll 67, the sheet passes over a roll 68 and is then wound and packaged.

While Figure V refers to the combining of two 0.381 mm sheets to form a 0.762 mm composite, it is apparent that other sheet thicknesses can be used. In addition, wide variation in sheet surfaces and sheet composition is permissible as well as wide variations in the printing of the sheets. In regard to the latter, one or both sheets may be printed with any desired patterns, designs or colors.

One of the major problems in sheet combining is wrinkling of the sheet in both the machine direction and cross-machine direction. These wrinkles are caused by sheet growth on the combining rolls during heating on the roll and by vapors released from the surface of the sheet which are trapped between the sheet and the roll surface behind the combining nip. The wrinkles caused by sheet growth during heating can be controlled by restricting the temperature rise of the sheet on the combining roll to less than 17°C by preheating the sheet before it gets to the combining roll or by introducing machine direction and cross-machine direction stresses into the sheet before it reaches the combining rolls. By preheating within 11°C of the sheet combining temperature, the sheet growth on the combining rolls is limited to an amount insufficient to cause wrinkles. A combination of the two techniques is preferred.

In addition, the tension, both in the machine and cross-machine directions, the temperature and the line speed of the sheets to be combined should be as evenly matched as possible.

Figure VI illustrates a sheet combining process similar to that shown in Figure V above, except that several tension, catenary loop and temperature controls have been added. These process controls are designed to match the tension, temperature and line speed of the sheets to be combined so as to provide optimum properties in the resulting composite interlayer.

Clear sheet 80 enters the upper section of the combining unit through a first upper tension nip 82. This tension nip controls the sheet tension through the pre-conditioning section comprising pre-conditioning heaters and a dewrinkling section where the sheet is dewrinkled using conventional means. Sheet tension in the pre-conditioning section is controlled at a minimum level by a first upper tension sensor 83.

A first upper temperature sensor 84 controls the pre-conditioning heaters to obtain

69, the combined sheet (0.762 mm) is to 82°C with 43°C to 71°C being preferred.

The clear sheet then passes through a second upper tension nip 85 into a relaxing section where the sheet is allowed to droop in a controlled catenary loop between the second upper tension nip 85 and the third upper tension nip 88. Tension nips 85 and 88 control the tension and catenary loop in the relaxing section. The speed of these tension rolls, which form tension nips 85 and 88, is controlled by the catenary loop sensor 86. The catenary loop is maintained in the relaxing section to relax out as much inherent sheet stress as possible so that the clear sheet, which enters this section with a shrink level of from 4% to 24%, leaves with a shink level of less than 4%. Also included in the relaxing section is second upper temperature sensor 87.

The third upper tension nip 88 controls the tension through the sheet stressing section to achieve drawdown levels in the range of from 1% to 30% with 4% to 16% being preferred. This nip is controlled by a second upper tension sensor 89.

After the relaxing section the sheet passes through a heating section where the sheet temperature is again adjusted in the range of from 32°C to 82°C, with 43°C to 71°C being preferred.

The clear sheet 80 then passes over a lay-on roll 90 onto a temperature control roll 91 in order to adjust the sheet temperature to the desired combining temperature. The temperature of the sheet just before combining is measured and controlled by a third temperature sensor 93, which is located just before the combining nip 94. After leaving the temperature control roll 91, the clear sheet 80 passes over an upper flexible bowed roll 92 for dewrinkling and then passes into the combining nip 94.

Meanwhile, polyvinyl butyral sheet 81, which has been printed with a color gradient, enters the lower section of the sheet-combining unit which is essentially the same as that described above.

The printed sheet 81 enters the lower section of the combining unit through a first lower tension nip 95 and then through a pre-conditioning section which comprises pre-conditioning heaters and a dewrinkling unit. This section may not be needed in an inline operation where the sheet is conditioned to a certain temperature and dewrinkled just prior to printing and then fed directly into the combining unit. Also located in the pre-conditioning unit is a first lower tension sensor 96 and a first lower temperature sensor 97. The printed sheet 81 then passes into a relaxing section formed by a second lower tension nip 98 and a third lower tension nip 101. In the relaxing section the sheet is allowed to droop in a controlled catenary loop where a lower catenary loop sensor 99 and a second lower temperature sensor 100 measure the droop and the temperature of the sheet, respectively.

Upon leaving the third lower tension nip 101, which is controlled by a tension sensor 102, the sheet passes into a preheating section similar to that discussed above. The sheet which is preheated as in the upper section, passes over a lay-on roll 103 onto a lower temperature control roll 104 and then over a lower flexible bowed roll 105 into the combining nip 94. A third lower temperature sensor 106 is located between the flexible bowed roll 105 and the combining nip 94.

Referring again to Figure VI, a control section 109 is provided to receive tension, temperature and catenary loop data from the sensors in the upper and lower section and to adjust these variables so that the tension, temperature and degree of relaxation in the catenary loop is matched for the upper and lower sections so as to provide optimum properties in the laminate.

In the combining nip 94 the clear sheet 80 is press-tacked to the printed sheet 81 so as to form a composite where the printed surface is at the interface of the two sheets.

After combining, the composite sheet 108 passes over a number of cooling rolls 107, which reduce the sheet temperature to less than 27°C with a temperature of less than 21°C being preferred. The composite sheet is then wound and transferred to shipping or storage.

The roll speeds, nip pressures and temperatures used in reference to the process illustrated in Figure VI are the same as those given in reference to the process illustrated by Figure V unless otherwise specified.

The above description of Figure VI refers to an upper and lower section. However, it is understood that the operation can be carried out side by side in which case the upper and lower sections would become the left and right sections or first and second sections of the operation.

It is also possible to combine two printed sheets using the above processes.

In the case of a three ply composite the middle layer can be printed on both sides and then laminated between two clear sheets, using modifications of the processes outlined above.

The heating steps referred to throughout the specification may be carried out by those means that are well known in the art. The sheet may be heated by any of

those various means which include, for example, electrical heaters, IR heaters, hot air devices, steam heated devices and hot water heated devices. Any cooling steps which are needed to adjust sheet temperature can use conventional means such as cool air or chill rolls. The degree of wrap around any given roll may be varied within broad limits.

The composites of the present invention are usually found to have a bond strength, as measured by a 180 degree pull apart test, of at least 0.295 Kg/cm. Such a bond should be sufficient to maintain the unitary nature of the composite during shipping, storage, handling and fabrication into laminated safety glass. During the glass laminating step the heat and pressure used to make the laminate also serve to increase the bond strength of the composite interlayer.

Preferably, the composite sheet has a curl value of less than 20%. The curl value is measured by cutting a sample approximately 508 cm x 2 cm from the machine direction of composite. The test sample is then placed in a circulating air oven maintained at 43°C. ± 1°C. for ten minutes. In the oven one-half of the test sample is supported on a flat shelf while the other half hangs down unsupported. The sample is then removed from the oven after 10 minutes and placed on a flat table with the surface of the sheet that was supported in the oven placed face down on the table. The sample is examined and if the ends of the sample curl back from the table surface, the distance from the end of the sample to the point that is still in contact with the table is measured in millimeters. The measurements for each end of the sample are averaged and the percent curl is determined by the following formula:

mm of curled sheet/original length (millimetres) X 100 = % curl.

A curl value greater than 20% indicates that there are residual stresses in the sheet which will cause the sheet to curl thereby causing processing difficulties in the glass laminating operations. More preferably, the composite sheet has a curl value of less than 15% and most preferably, less than 10%.

A 30 mil. composite gradient sheet prepared by the process illustrated in Figure VI, having a bond strength of 0.295 Kg/cm and a curl value of 3% is laminated between two sheets of glass of the type conventionally used in the preparation of automobile windshields. The resulting laminated safety glass is evaluated for optical density, sheet beauty and adhesion and compared to glass laminates prepared using a single ply 0.762 mm polyvinyl butyral interlayer prepared by conventional methods. The properties of the glass laminates prepared using the composite interlayer of the present invention are comparable to the properties of those laminates prepared using a conventional single ply polyvinyl butyral interlayer.

WHAT WE CLAIM IS:

1. A composite suitable for use as an interlayer for laminated safety glass, which composite comprises at least two sheets of thermoplastic polymeric material combined together in face-to-face contact with at least a portion of at least one of the sheets being printed with an ink on a side of the sheet that is in face-to-face contact with another of the said sheets, the thermoplastic polymeric material being polyvinyl butyral, a polyurethane, a polyamide, poly(ethylene-vinyl acetate) or poly(ethylene-vinyl alcohol).

2. A composite according to Claim 1 in which the thermoplastic polymeric material is polyvinyl butyral.

3. A composite according to Claim 2 wherein one of the sheets of polyvinyl butyral is printed with a color gradient.

4. A composite according to either of Claims 2 and 3 having a total thickness in the range of from 0.51 to 1.91 mm.

5. A composite according to any of Claims 2 to 4 wherein two sheets of polyvinyl butyral are combined.

6. A composite according to any of Claims 2 to 4 wherein three sheets of polyvinyl butyral are combined.

7. A composite according to Claim 6 wherein the middle sheet of polyvinyl butyral is printed on both sides.

8. A composite according to Claim 1 substantially as described herein with reference to any of Figures I to III of the accompanying Drawings.

9. Laminated safety glass comprising a composite according to Claim 1 between two sheets of glass.

10. Laminated safety glass comprising a composite according to any of Claims 2 to 8 between two sheets of glass.

11. A process for preparing a composite according to any preceding claim, which process comprises:

A. passing a first continuous sheet of a thermoplastic material which is polyvinyl butyral, a polyurethane, a polyamide, poly(ethylene-vinyl acetate) or poly(ethylene-vinyl alcohol) between nip rolls to impart machine direction, sheet tension and drawdown into the sheet;

B. adjusting the temperature of the sheet to a temperature in the range of from 32 to 82°C;

C. removing any wrinkles from the sheet; and then

D. combining in face-to-face contact the first continuous sheet with a second

continuous sheet of thermoplastic material which is polyvinyl butyral, a polyurethane, a polyamide, poly(ethylene-vinyl acetate) or poly(ethylene-vinyl alcohol) wherein the second sheet has received substantially the same treatment as outlined in process steps A to C above, at least a portion of at least one of the sheets being printed with an ink on a side of the sheet that is brought into face-to-face contact with the other sheet; and

E. cooling the resulting composite.

12. A process according to Claim 11 wherein the thermoplastic sheets are polyvinyl butyral.

13. A process according to either of Claims 11 and 12 wherein the second continuous sheet is printed with a color gradient.

14. A process for preparing a composite suitable for use as an interlayer for laminated safety glass, which process comprises extruding a layer of molten polyvinyl butyral on to the surface of a moving web of polyvinyl butyral, which surface has been previously printed with a color gradient.

15. A process for preparing a composite suitable for use as an interlayer for laminated safety glass, which process comprises:

A (1) passing a first continuous sheet of polyvinyl butyral between a first set of nip rolls to impart machine direction, sheet tension and drawdown into the sheet;

(2) adjusting the temperature of the first sheet to a temperature in the range of from 32 to 82°C. while removing any wrinkles in the sheet;

(3) allowing the first sheet to assume a catenary profile between second and third sets of nip rolls;

(4) adjusting the temperature of the first sheet to a temperature in the range of from 32 to 82°C; and simultaneously with steps 1 to 4 above;

B. (1) passing a second continuous sheet of polyvinyl butyral which is printed on one side with a color gradient, between a fourth set of nip rolls and

(2) allowing the second sheet to assume a catenary profile between fifth and sixths sets of nip rolls;

(3) adjusting the temperature of the second sheet to a temperature in the range of from 32 to 82°C; and

C. (1) passing the first and second sheets into a combining nip where they are combined wherein the printed surface of the second sheet of polyvinyl butyral is combined to the first sheet so as to provide a composite wherein the bond strength between the first and second sheet is at least 0.295 Kg/cm;

(2) cooling the resulting composite;

wherein the process steps outlined in A and B above are controlled to provide substantially the same line speed, sheet tension and temperature in each of the first and second sheets of polyvinyl ana9) fed into the combining nip.

16. A process according to Claim 15 wherein the shrink level of the first and second sheets is reduced to less than 4% prior to entering the combining nip.

17. A process according to either of claims 15 and 16 wherein the resulting composite has a curl value as hereinbefore defined of not less than 20%.

18. A process according to any of Claims 15 to 17 wherein the temperature rise of the first and second sheets during the combining step is less than 17°C.

19. A process according to any of Claims 15 to 18 wherein the sheet tension, temperature and line speed for each of the first and second sheets are constantly monitored and synchronized.

20. A process for preparing a composite suitable for use as an interlayer for laminated safety glass, which comprises combining a sheet of polyvinyl butyral which has been printed on both sides with a color gradient between two other sheets of polyvinyl butyral by passing the sheets through a combining nip; wherein the temperature, tension and line speed of each of the sheets, prior to entering the combining nip, is substantially the same.

21. A process for the production of a composite substantially as described herein with reference to any one of Figures IV, V and VI of the accompanying Drawings.

22. A composite prepared by a process according to any of Claims 11 to 21.

23. Laminated safety glass comprising a composite according to Claim 22 as an interlayer between two sheets of glass.

J. C. LUNT
Chartered Patent Agent,
Monsanto House,
10-18 Victoria Street,
London, SW1H 0NQ.

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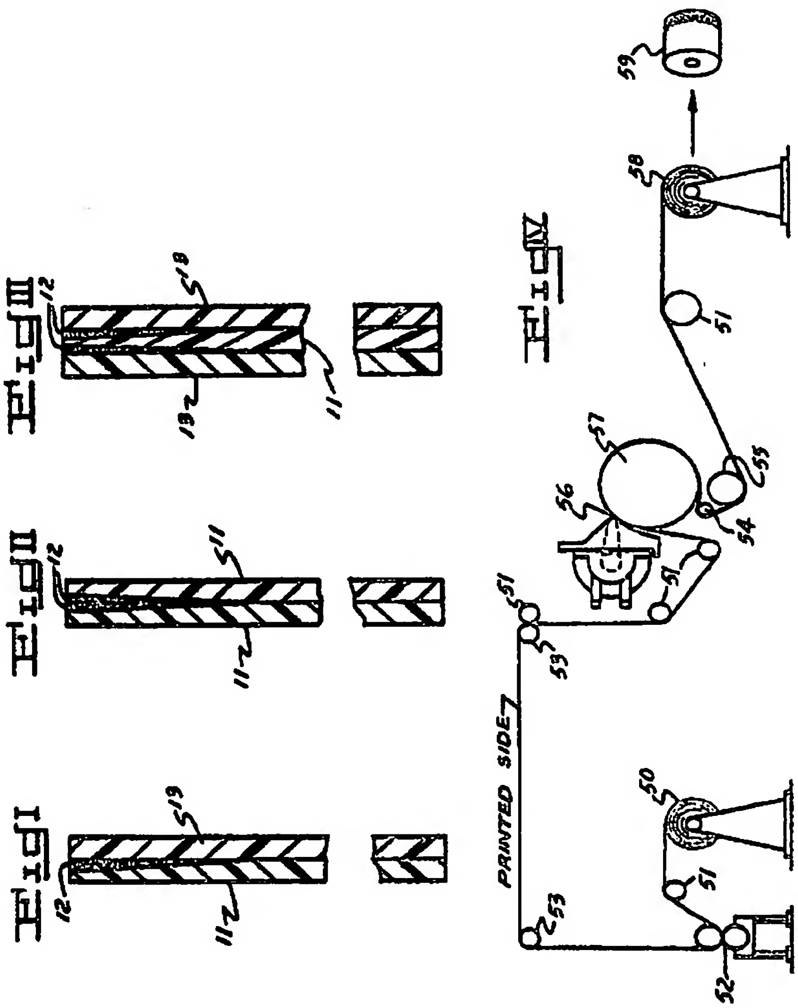
FIG. I
FIG. II
FIG. III
FIG. IV
PRINTED SIDE
11
12
13
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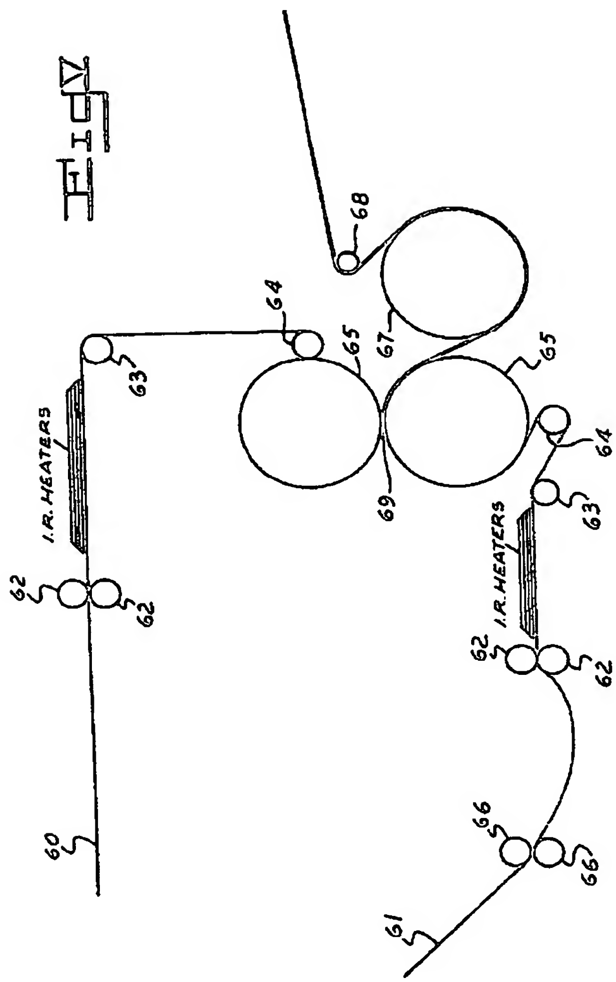
1470844 COMPLETE SPECIFICATION

3 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 2

FIG. V



1470844 COMPLETE SPECIFICATION

3 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 3

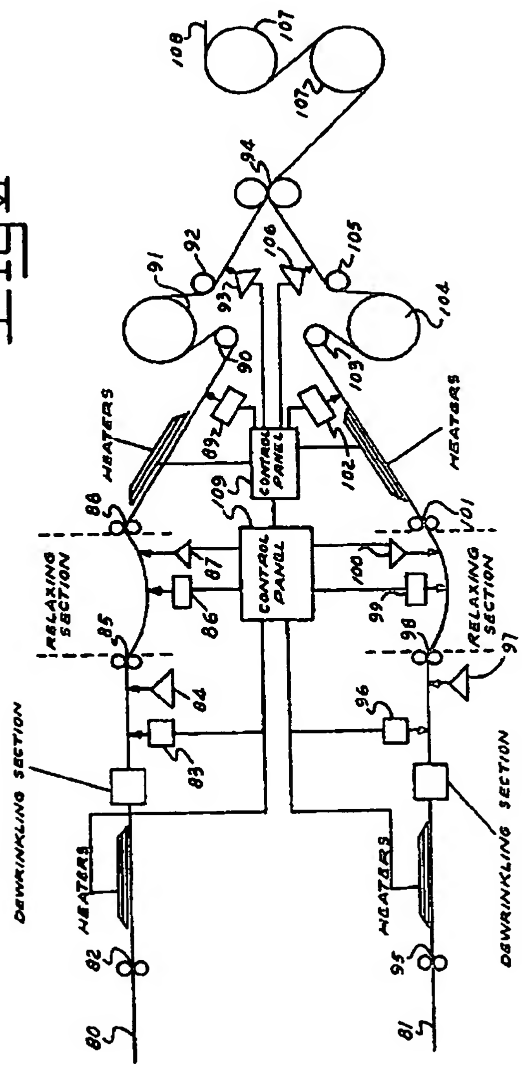


FIG VI